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NON-DESTRUCTIVE MECHANICAL CHARACTERISATION OF MECHANICAL PROPERTIES OF NON-HOMOGENEOUS NANOSTRUCTURED COATINGS

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Abstract

The experimental model permitting the detection of coating non-homogeneity is suggested for standard depth sensing indentation tests.

It is assumed that the shear modulus varies arbitrarily with depth in a nanostructured coating. It is defined a notion of the stiffness as a function of radius of a contact zone for nanostructured coating coupled with homogeneous half-space, which makes it possible to classify the non-homogeneity of a coating.

Using numerical examples, the influence of different laws of shear modulus variation in a coating on the stiffness is studied.

1. Introduction

The protection of materials by means of non-homogeneous coatings is now a well-established technology, and is an extremely versatile means of improving component performance [1]. Coating elasticity is a crucial parameter for the performance and reliability of a coated part. The determination of mechanical properties arises when one wants to control the coating quality.

At present nano-indenters are high-precision instruments which allow the performance of non-destructive tests on thin non-homogeneous coatings. But the interpretation of such test results is still under development. In this paper a mathematical simulation of standard nano-indentation tests is presented. The predictions from this simulation for different non-homogeneous coatings are reported.

The elastic contact problem, which plays a key role in the analysis procedure, was originally considered in the late 19th century by Boussinesq [2] and Hertz [3]. Hertz analysed the problem of the elastic contact between two spherical surfaces with different radii and elastic constants. His now classic solutions form the basis of much experimental and theoretical work in the field of contact mechanics.

In the early 1980's, it was realised that load and depth sensing indentation methods could be very useful in the measurement of the mechanical properties of very

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thin films and surface layers, and instruments for producing submicron indentations were developed.

Oliver, Hutchings, and Pethica [4,5] suggested a simple method based on measured indentation load-displacement curves and knowledge of the indenter area function (or shape function); that is, the cross-sectional area of the indenter is related as a function of the distance from its tip.

The shortcoming of these methods is that precise evaluation of the elastic modulus is performed only for samples with constant elastic modulus at depth. In the case of a non-homogeneous coating coupled with a homogeneous half-space, the obtained modulus is some mean of both the elastic modulus of a coating and the elastic modulus of a substrate.

Suresh et al. [6] proposed the method based on the finite element simulation for estimation of Young's modulus variations through a compositionally graded layer by recourse to spherical indentation.

The problem of properties investigation of non-homogeneous materials attracts the attention of many researchers by its actuality and complexity, mathematically as well as experimentally [6,7]. Published solutions of a contact problem for non-homogeneous materials were constructed in most cases numerically (using finite element method).

In this work the authors suggest the value of the numerical-analytical method. This method makes it possible to more deeply analyze the cause of problems which arise in investigation of mechanical properties of non-homogeneous nanostructured materials. Derived analytical expressions, which define the solution of the contact problem for a non-homogeneous coating coupled with homogeneous half-space degenerate into the classic Hertzian solution in the case of homogeneous half-space. In numerical examples, the stiffness of a material has been considered and analysed. The relation between the law of shear modulus variation with depth and the stiffness of a material as a function of radius of a contact zone is presented.

For the determination of properties of a non-homogeneous coating the size of contact zone of an indenter with a coating is an essential value in contrast to that of a homogeneous material. In this paper it is shown that the experiment which is performed with a view to investigate the elastic properties of a non-homogeneous coating should at least be carried out for contact zones commensurate with the thickness of a coating (from $1/4\ T$ to $4T$, here T is the thickness of a coating).

To analyse the non-homogeneous character of a nanostructured coating it is necessary to determine the dependence of the stiffness value from the size of a contact zone for the wide range of its variation. However an experiment should be carried out within the limits of the linearly elastic deformation of a material. Depth sensing indentation tests using the set of spherical indenters with different diameters settle this problem.

2. Formulation of the contact problem simulating an indentation test

A non-deformable spherical indenter of radius R is impressed into a surface Γ, of a non-homogeneous elastic half-space Ω, by a normal force P (Fig. 1). Cylindrical (r, φ, z)

coordinates are bound to the half-space. It is assumed that all deformations are elastic and that the size of the contact zone, a, is small with respect to the radius R of the sphere, and that friction force does not exist between the indenter and the surface of half-space.

The spherical indenter surface is approximated by a quadratic shape $z = \psi(r) = \beta r^2$ in the vicinity of the original point of contact. This approximation is valid for small contact radii, $a<0.1R$ for homogeneous solids [6], which covers essentially all practical cases of elastic spherical indentations.

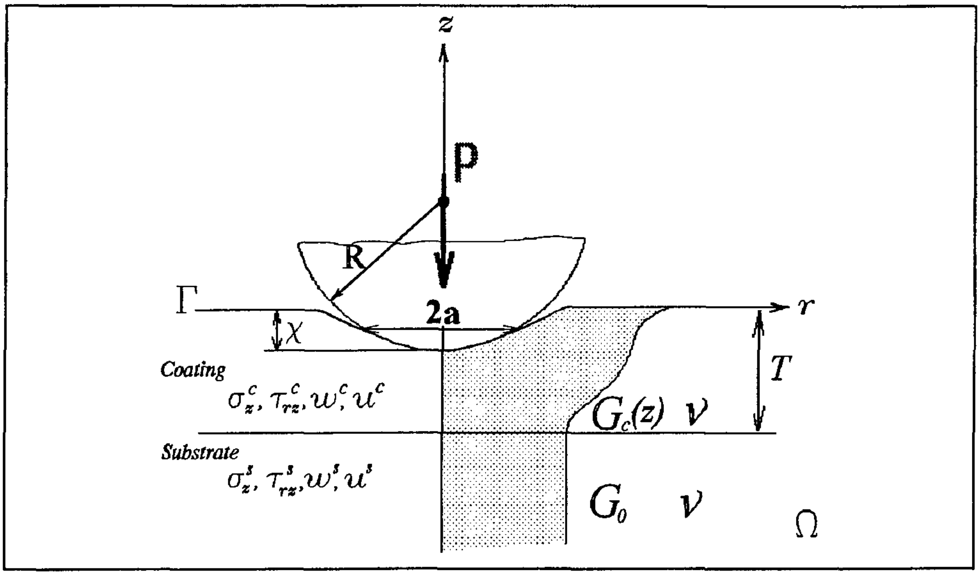

Figure 1. Scheme of indentation test.

The half-space is not loaded outside the indenter. Under the action of the normal force, P, the indenter moves a distance χ along the z axis.

We assume a Poisson ratio, ν is a constant, and consider the shear modulus, $G(z)$, in the half-space to exhibit a well defined variation. The shear modulus can thus be arbitrarily continuous or a piece-wise continuous function of the depth z, as thus expressed as follows:

$$\begin{aligned} &1.\ G(z) = G_c(z), && -T \leq z \leq 0 \\ &2.\ G(z) = G_c(-T) = G_0, && -\infty < z \leq -T \end{aligned} \tag{1}$$

Let us denote $G_c(0)=G_1$. Under the above assumption, the boundary conditions have the form:

$$z=0:\ \tau_{zr} = \tau_{z\varphi} = 0,\quad \sigma_z = 0,\quad r > a, \tag{2}$$
$$w = \chi(r) = \chi - \psi(r),\qquad r \leq a$$

Here w is the displacement along the z axis, while τ_{zr}, $\tau_{z\varphi}$, and σ_z are the radial, tangential and normal stresses respectively.

Using the method of works [8,9,10], the solution to the contact problem was constructed and the relation between an impressing force and the size of a contact zone was determined [11].

$$P = 4\frac{a^3}{R}\frac{G_1}{1-\nu}\left(\frac{2}{3}\frac{G_0}{G_1} + \sum_{i=1}^{N} C_i\left(-\cosh a_i + a_i^{-1}\sinh a_i\right)\right).$$

Here C_i and a_i are certain constants defined by non-homogeneity laws.

3. The Stiffness Concept of the Depthwise Non-homogeneous Material

As a result of the penetration of the indenter into the non-homogeneous material we can obtain the relation between the impressing force and the displacement of the indenter. These values by themselves are not very informative, as it is difficult to use these values to determine the existence of the non-homogeneous layer on the surface of the foundation.

We define an expression which is referred to as the stiffness of the material

$$S = \frac{3}{4}\frac{P}{a\chi(1-\nu^2)},$$

where a is the contact zone radius, χ is the displacement of the indenter, ν is the Poisson's ratio. For the homogeneous material the stiffness is a constant, equivalent to the shear modulus of the foundation [12].

For the non-homogeneous material, $S(a)$ is a function dependent upon the size contact zone.

4. Testing method for determination of the non-homogeneity of a material

Our aim is to detect the character of the non-homogeneity of a material by using standard indentation measurements.

The method described here is based on a spherical indenter and aims at investigating the elastic properties of materials. Thus, the test must be carried out under elastic deformation only. This is the basis of the method, and it allows the depth distribution of material properties to be investigated.

Practically all existing materials deform elastically in a narrow range of loading. One of the starting problems is the determination of that range.

The goal of the test is to determine the force-displacement relation for the penetration of a non-deformable spherical indenter into the continuously non-homogeneous coating coupled with a homogeneous half-space. To guarantee the non-

destructive character of the test, it is necessary to fulfil the following condition : the stresses must not exceed the elasticity limits.

5. Numerical Results

The numerical analysis has been carried out for non-homogeneous nanostructured coatings which have applications in magnetic recording systems, printing industries and motor engines. In this case, the value of the ratio of the shear modulus on the coating surface to the shear modulus in any interior point of substrate (denoted n) was bounded such as

$$1/3.5 < n < 3.5$$

Shear modulus is assumed to vary with depth, according to the relation

$$G(z) = \begin{cases} G_0 f_i(z) & -T \leq z \leq 0, \\ G_0 & z \leq -T, \quad i = 1,2,3,4 \end{cases} \tag{3}$$

$$f_1(z) = 3.5, \qquad f_2(z) = \frac{1}{3.5},$$

$$f_3(z) = 3.5 + 2.5\frac{z}{T}, \quad f_4(z) = \frac{1}{3.5} - \frac{2.5}{3.5}\frac{z}{T}$$

Fig. 2 shows the values $f_i(z')$, $z' = z/T$ (i = 0,1,2,3,4) which characterise non-homogeneity laws described above.

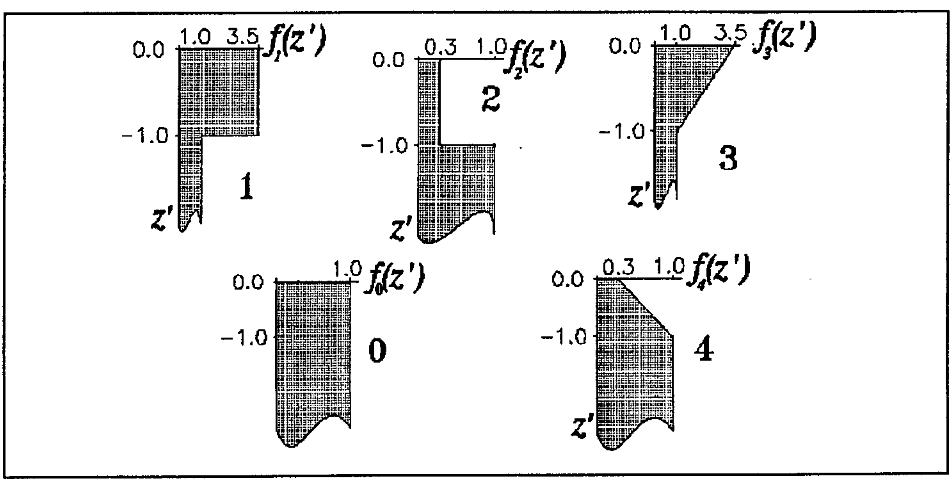

Figure 2. Nonhomogeneity laws describing the variation of shear modulus with depth.

Fig. 3 shows graphs $S(\lambda^{-1})/S_0$ ($\lambda=T/a$) — the ratio of the stiffness of non-homogeneous coatings, S, to the stiffness of substrate — a homogeneous half-space, S_0 for the 5 cases given in Fig. 2. To make the graph more descriptive we present them using a logarithmic scale. The curve numbers correspond to the variation laws of elasticity modulus.

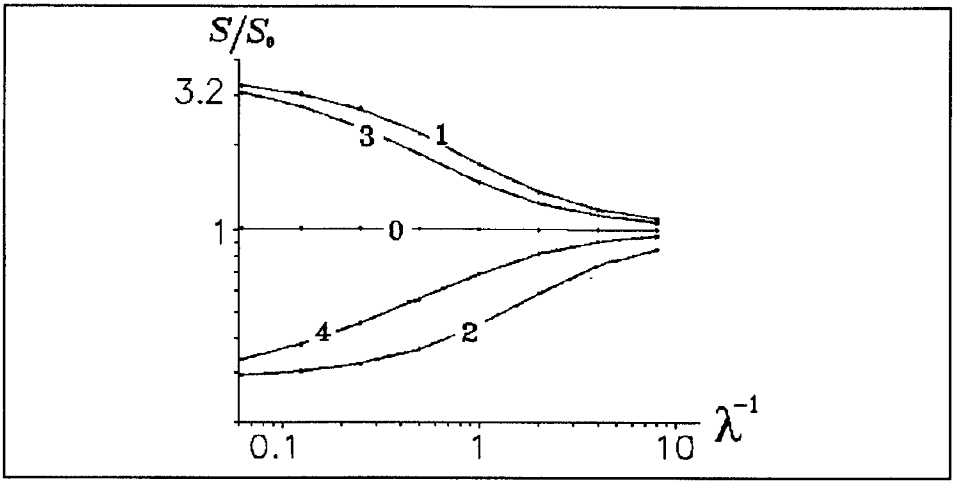

Figure 3. Graphs of the ratio $S(\lambda^{-1})/S_0$ for the cases shown in Fig *2.*

Fig. 3 shows that by using the results of non-destructive indentation experiments we can evaluate the variation of elasticity modulus with depth. Moreover, it shows that this kind of test gives the possibility of distinguishing changes in surface layer properties not only in terms of being softer or harder, but also in terms of the variation of elastic properties of coatings with depth (blended or layered). A potential application of interest is the characterisation of compositionally modulated multilayers in microelectronics [13].

The values on the graphs are obtained from calculations in which the contact zone, *a,* is varied from *1/16 T* to *16 T.* This is practically impossible to reproduce in one experiment within elastic deformations limits. Hence, the solution of the problem of extrapolation of values obtained for the narrow interval of indenter penetration values and, respectively, of contact zones on the whole domain of definition is of great interest.

It is obvious from Fig. 3, that the middle region of the stiffness change graph is the most informative ($1/4T \leq a \leq 4T$).

6. Conclusion

In this work the estimation of relevant parameters for the non-destructive testing of the mechanical properties of non-homogeneous coatings has been carried out. The method

is based on a depth sensing indentation test performed within the elastic limits of the tested coated materials.

The method presupposes the mathematical simulation of the testing process that involves the penetration of spherical indenter into a functional non-homogeneous coating with an arbitrary variation of the elasticity modulus along its depth.

This method permits the interpretation of indentation test results performed in the elastic deformation region on non-homogeneous coatings by existing standard equipment.

From the point of view suggested in this paper regarding the testing method for determination of non-homogeneity of a coating, we analysed similar experimental data presented in the paper of Suresh et al. [6]. In this work the authors considered two kinds of coatings which correspond with above mentioned laws 3 and 4 — linear increasing and linear decreasing laws. The authors simulated a non-homogeneous coating by the model of a material with elastic properties varying exponentially with depth. Presented data correspond to small values of a contact zone in our terms, e.g. $\lambda_1 > 29.2$, $\lambda_2 > 13.3$ (here $\lambda = T/a$, $T_1 = 2.92$ mm, $T_2 = 1.33$ mm, $a < 0.1$ mm). Such values of λ conform to the very beginning of the stiffness curve and correspond with properties of practically homogeneous material and reflect properties only of layers attached to the surface layer. In other words, the experimental data do not give the opportunity to observe the non-homogeneity variation with depth because of small variation of a contact zone.

The main conclusion of our work is that in the investigation of non-homogeneous coatings, the size of a contact zone should vary at least up to the value comparable with the thickness of coating.

7. References

1. Rickerby, D. and Matthews, A. (1991) *Advanced Surface Coatings*, A Handbook for Surface Engineering , Blackie, London.
2. Boussinesq, J. (1885) *Applications des Potentiels a l'etude de equilibre et du mouvement des solides elastiques*, Gauthier-Villars, Paris.
3. Hertz, H. (1882) Uber die Beruhrung fester elastischer Korper (On the contact elastic solids), *J. reine und angewandte Mathematik* **92**, 156-171.
4. Oliver, W.C., Hutchings, R. and Pethica, J.B. (1986) In ASTM STP **889**, edited by Blau and B.R. Lawn (American Society for Testing and Materials), Philadelphia. PA., 90-108.
5. Pharr G.M. and Oliver W.C. (1992) Measurement of thin film mechanical properties using nanoindentation, *MRS Bulletin* 7 , 28-33.
6. Suresh, S., Giannakopoulos, A.E. and Alcala, J. (1997) Spherical indentation of compositionally graded materials: theory and experiments. *Acta mater.* **45**, No 4, 1307-1321.
7. Anderson, I.A. and Collins, I.F. (1995) Plane strain stress distributions in discrete and blended coated solids under normal and sliding contact. *Wear* **185**, 23-33.

8. Aizikovich S.M., Aleksandrov V.M. (1984) Axisymmetrical Problem about Indentation of Round Punch into Elastic Inhomogeneous with Depth Half-Space. *Izv. Akad. Nank SSSR. Mech. Tverd. Tela* **2**, 73-82
9. Aizikovich, S.M. (1990) An asymptotic solution of one class of dual equations for small values of the parameter, *Doklady Akademii Nauk SSSR* **313**, No. 1, 48-52.
10. Aizikovich, S.M. (1992) An asymptotic solution of one class of dual equations for large values of the parameter, *Doklady Akademii Nauk SSSR* **319**, No. 5, 1037-1041.
11. Aizikovich S.M., Krenev, L.I. and Serova, N.A. (1998) Non-Destructive Determination of Mechanical Properties of Non-Homogeneous Coatings. *7-th European Conference on Non-destructive Testing*, Copengagen, Denmark, May 26-29, Proceedings **1**, 1063-1069.
12. Johnson, K.L. (1985) *Contact Mechanics*, Cambridge University Press, Cambridge.
13. Haseeb, A., Celis, J.-P. and Roos, J.P. (1995) An Electrochemical Deposition Process for the Synthesis of Laminated Nanocomposites, *Materials Manufacturing Processes*, **10**, (4), 707-716.